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Density Determination of Silver Neodecanoate, Tungsten Hexacarbonyl, and a series of Metal Acetylacetonates and Hexafluoroacetylacetonates.

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Abstract

The densities of a series of metal-organic compounds potentially suitable for metal-organic chemical vapor deposition, MOCVD, formation of metal interconnection films, were determined using a gas pycnometer which measures the pressure change when a gas(helium) is allowed to expand in the presence of a solid material whose density is to be determined. The solid material is contained in a calibrated chamber. Using the ideal gas equation, the volume of the solid material can be calculated, and thus it's density. Densities are reported for silver neodecanoate, tungsten hexacarbonyl, and metal acetylacetonates and hexafluoroacetylacetonates of aluminum, chromium, cobalt, copper, magnesium, nickel, palladium, platinum, rhodium, and zinc.

Key words: density, silver neodecanoate, tungsten hexacarbonyl, metal acetylacetonates, metal hexafluoroacetylacetonates, acetylacetonates, hexafluoroacetylacetonates, MOCVD of metals, density of metal-organic compounds.

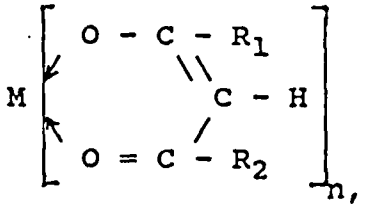
Introduction

Studies of thin film preparation alternatives for the microelectronics industry have prompted examination of the physical, thermal and optical properties of a series of metal-organic compounds which have received limited characterization in the literature[1]. This paper reports on the densities of a series of these compounds currently under investigation in our laboratories. In general, these compounds possess desirable properties which make them potentially viable alternatives to the conventional, more costly methods for deposition of pure metal, alloy, or insulator films. They also offer alternatives to lithographic patterning processes currently used in microelectronics. These properties are 1) most of them decompose at low temperatures(as low as 150-200°C) in either the solid or vapor phase; 2) they are easily handled solids at room temperature; 3) some may be photoactive; 4) they are soluble in a wide variety of solvents and 5) many, if not all of them exhibit appreciable vapor pressures at temperatures below that at which decomposition is significant.

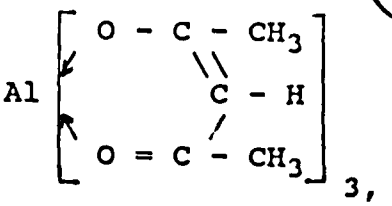
In general, the acetylacetonates decompose in the solid state at relatively low temperatures, but still exhibit appreciable vapor pressures at temperatures below which

their decomposition rate is significant. Hexafluoro derivatives of these compounds are in general more volatile and appear to achieve atmospheric pressure as solids, or more generally, as liquids before they begin to decompose. Those of interest for metal film formation may also be attractive candidates for metal interconnection pattern repair for which there is no usable technology(metal-organics have been used in metal lithographic mask repair[2,3]).

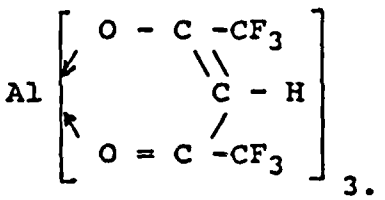
Present research in these laboratories is involved with a series of metal-organic compounds called 1,3-diketonates, commonly called acetylacetonates, or acacs. These compounds have the general formula,



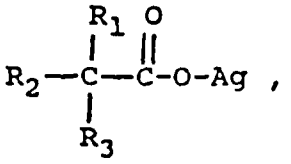
where M is the metal in a +n oxidation state. In our studies, R_1 and R_3 are either methyl or fluorine substituted methyl groups. An example of two such compounds are aluminum(III) acetylacetonate,



and aluminum(III) hexafluoroacetylacetonate(hfa),



Other compounds under investigation are silver neodecanoate, $AgOOCC_9H_{19}$,



and tungsten hexacarbonyl, $W(CO)_6$. With the exception of $Al(acac)_3$[4], $Cu(acac)_2$[5], and tungsten hexacarbonyl[3], no information could be found regarding the density of the metal-organic compounds. In an earlier paper[1], we used a different technique to measure the density of rhodium(III) acetylacetonate. This latter, more conventional method is compared with the present approach. Densities are reported for $Al(acac)_3$, $Cr(acac)_3$, $Co(acac)_3$, $Co(acac)_2 \cdot H_2O$, $Cu(acac)_2$, $Mg(acac)_2\ 2H_2O$, $Ni(acac)_2 \cdot xH_2O$, $Pd(acac)_2$, $Pt(acac)_2$, $Rh(acac)_3$, $Zn(acac)_2 \cdot xH_2O$, $Al(hfa)_3$, $Cr(hfa)_3$, $Co(hfa)_3$, $Cu(hfa)_2 \cdot 2H_2O$, $Mg(hfa)_2 \cdot 2H_2O$, $Ni(hfa)_2 \cdot 2H_2O$, $Pd(hfa)_2$, $Pt(hfa)_2$, $Zn(hfa)_2 \cdot 2H_2O$, silver neodecanoate, and tungsten hexacarbonyl.

Experimental

A. Materials

The metal acetylacetonates and hexafluoro-acetylacetonates were purchased from Strem Chemicals Inc.(7 Mulliken Way, Newburyport, Ma. 01950). Silver neodecanoate was purchased from Electrink, Inc.(7414 Trade St., San Diego, Ca. 92121), and tungsten hexacarbonyl from Alpha Products(152 Andover St., Danvers, Ma. 01923). The helium used in density measurements was supplied by Airco(P.O.B. 12338, RTP, N.C. 27709) as a 99.9995% pure product. The metal-organics were stored in the containers in which they were received, in a desiccator over a desiccant, (TM)Drierite(Fisher Sci. Co., Raleigh Branch Office, P.O. Box 40339, Raleigh, N.C. 27629).

The metal-organics studied were analyzed by Galbraith Laboratories Inc.(P.O.B. 51610 Knoxville, Tn 37950). The metal content was determined by atomic absorption and the carbon and hydrogen analysis was determined by combustion analysis. The results are tabulated in Table I. Two compounds, the nickel and zinc acetylacetonates, were found to be out of the acceptable range for purity. The nickel compound, for example, shows good correlation with the expected analysis for the monohydrate, as far as carbon and hydrogen are concerned. However, the metal composition correlates with the anhydrous composition. The zinc compound

has similar problems. For example, the carbon composition is neither that of the di, mono, or anhydrous material while the hydrogen content is close to the anhydrous. The metal content is similarly close to the anhydrous value. These compounds could not be obtained in purer form. Recrystallization from methanol did not rectify the stoichiometric discrepancies. Consequently, the densities determined for the $Ni(acac)_2$ and $Zn(acac)_2$ must be viewed in this light. An attempt was made to synthesize them in our laboratory by the method described by Charles and Pawolskwi[6]. The resulting material gave analyses which were as confusing as the commercial products described in Table I. The zinc and nickel acacs have been studied by Cotton [7,8,9] and were reported to exist as tetramers and trimers in the solid state which might account for the difficulty in preparing them in stoichiometric form.

The $Co(acac)_2$, as based on analysis, is seen from Table I to be a monohydrate, although the literature[6] only reports the anhydrous and dihydrate form of the compound. The reason for this seeming discrepancy, despite the excellent agreement between theory and actual composition of a monohydrate form is not understood. Based on other studies the nickel(II), cobalt(II), and zinc(II) acacs tend to decompose before they achieve their normal sublimation or normal boiling points[10]. Depending on the details of their vaporization behavior they may not be as useful for vapor phase stimulated decomposition as their hexafluoro

counterparts. There inclusion in the present work is because of their potentials for solid state stimulated decomposition(and perhaps under proper conditions vapor phase decomposition), and for the sake of completeness, even though their purity or level of hydration is unclear.

As received, several of the metal-organic compounds, discussed below, were analyzed and appear to be hydrated. These compounds were placed in a vacuum desiccator containing both sulfuric acid and Drierite for several days to determine whether or not they could be dehydrated by such a procedure. The compounds were placed in beakers which were in turn placed in the desiccator. The desiccator was first evacuated to the low mTorr range using a mechanical pump and then the dehydration, if any, was allowed to proceed. Analysis of the compounds indicated that they still contained water of hydration, although their stoichiometries may have changed slightly. The $Ni(hfa)_2$, $Zn(hfa)_2$, $Mg(acac)_2$, and $Mg(hfa)_2$ purchased as dihydrates remained essentially unchanged. The $Cu(hfa)_2$ which was purchased as an anhydrous compound analyzed as a monohydrate. It was also examined as described, and similarly, did not dehydrate under the conditions employed. If the copper compound is not protected from moisture under normal clean room ambient conditions, i.e. relative humidity 35%, it was found to hydrate further, to a dihydrate, with its color changing from a dark blue-green to a light green in the process. This

color change has also been observed by Bertrand and Kaplan[11].

Even though analyses were given to 2 decimal places by the commercial analytical laboratory used, it is felt that the experimental uncertainty exists in the first decimal place. Of the three elements analyzed for: carbon, hydrogen, and metal, it is seen from Table I that the best agreement between theory and the experimentally determined quantities was obtained for carbon. The hydrogen which is present in smaller concentrations shows greater deviation between theory and actual values, although in most cases it represents only a few percent deviation. Similarly, in some instances the metal concentration determined experimentally exhibits greater deviation from the theory than does the carbon data. This is attributed to the fact that the weight percent of metal is also fairly small, and susceptible to a larger percent error. The precision of the density measurements on these compounds, as shown in Table II, indicates that under the relative humidity conditions in the humidity controlled cleanroom in which the experiments were conducted, 35% RH and 22°C, the final hydrated compounds appeared stable during the course of the measurements of densities. However, as noted, the $Cu(hfa)_2$ compound hydrates further if exposed to the ambient atmosphere for a few days. To avoid problems, all the compounds were stored in sealed bottles in a desiccator between experiments.

B. Density Measurements

A (TM)Multipycnometer from Quantachrome Corp.(5 Aerial Way, Syosset, N.Y. 11791) was used for density measurements. A simplified schematic of the instrument is shown in Figure 1. The smallest sample cell, the microcell(cell volume of 12.416 cm^3), was used for density measurements. The powdered sample was packed in the sample holder as full as possible to minimize sample size contributions to non-reproducibility. For example, when the sample chamber was packed only half full, the measurement precision was poorer than when it was packed full. After each sample was loaded, a vacuum was drawn with a mechanical pump for two minutes, and then the system was purged with helium for an additional three minutes. Before any readings were taken, the sample cell was pressurized twice with helium to purge the system, and then vented to the air to relieve built up pressure. The density was then determined. It was found that reproducibility was critically dependent upon replacing all of the air in the system with the pycnometer fluid, helium. In this same vein, it was found that the vacuum step prior to introduction of the helium reduced the number of purges, or purge time required to obtain reproducible results. Three measurements were made on each sample, and all of the data for a given compound were averaged. The precision of each reading was within ±0.1% of the average. A standard, polycrystalline α-aluminum oxide(Fisher Sci. Co., Raleigh Branch Office, P.O. Box

40339, Raleigh, N.C. 27629) was used prior to and after each set of measurements to assure proper calibration of the instrument. All samples were weighed before and after the density measurements were taken to insure no material was lost during the vacuum step. The appendix gives a theoretical derivation of the working equation, based upon information supplied by the manufacturer.

Experimental Results and Discussion

Table II summarizes the densities of the metal-organic compounds. Values of the cell volumes, sample masses, and pressures were known to four significant figures. Published literature values of densities, where available, are shown in the third column. The values obtained in the present study appear to correlate well with the published literature values. From Table II it is seen that in all cases, the fluorinated acacs are denser than the simple acacs by as much as 50%. As mentioned, in the introduction, all of the compounds studied are soluble in a wide variety of solvents which makes the standard method of using a pycnometer with a liquid very difficult to implement. Although these compounds are generally insoluble in water which could be used conceivably for pycnometer measurements, as indicated, some of these metal-organics might tend to hydrate which would lead to erroneous results. A recent paper on the properties

of rhodium(III) acetylacetonate[1] by the present authors reported a density value of 1.75 g/cm^3. This number was determined in paraffin oil. Due to the high viscosity of paraffin oil, it was difficult to obtain reproducible readings which probably accounts for the approximately 8% discrepancy between the present and earlier work.

Compared with a liquid, a gas is better able to penetrate the finest pores in a powdered material, therefore assuring greater precision, and probably accuracy. Considering the small atomic dimension of helium, and its ideal gas behavior, its penetration into crevices and pores approaching 0.4-0.5 nm is possible. The excellent reproducibility of our data, and the agreement with the literature value for aluminum oxide, and the other compounds listed, attests to the efficacy of this technique. The aluminum oxide agreement is much better than 1%, as is the $Al(acac)_3$, similarly, for the $Cu(acac)_2$ and the $W(CO)_6$.

Conclusions

Density measurements of tungsten hexacarbonyl, silver neodecanoate, $Al(acac)_3$, $Cr(acac)_3$, $Co(acac)_3$, $Co(acac)_2 \cdot H_2O$, $Cu(acac)_2$, $Mg(acac)_2 \cdot 2H_2O$, $Ni(acac)_2 \cdot xH_2O$, $Pd(acac)_2$, $Pt(acac)_2$, $Rh(acac)_3$, $Zn(acac)_2 \cdot xH_2O$, $Al(hfa)_3$, $Cr(hfa)_3$, $Co(hfa)_3$, $Cu(hfa)_2 \cdot 2H_2O$, $Mg(hfa)_2 \cdot 2H_2O$, $Ni(hfa)_2 \cdot 2H_2O$, $Pd(hfa)_2$, $Pt(hfa)_2$, and $Zn(hfa)_2 \cdot 2H_2O$

are reported using a gas pycnometer. The latter relies on the pressure change that occurs when a gas(helium in this study), is allowed to expand in the presence of a solid material. This method is convenient, and rapid to use(20-30 min. per sample), and for material which is soluble in a wide variety of solvents, or which can solvate in these, this may be the only practical method. It has been found in the present study that reproducible results are obtained with small sample size(1-5 grams). The resultant density data correlate well with those reported in the literature for the few metal-organic compounds previously studied, and also for aluminum oxide which was used by us as a calibration standard. It was found that the hexafluoro-acetylacetonates were always denser than the simple acetylacetonates, by as much as 50%. It was also found that the hydrated forms of metal acacs and hfas could not be dehydrated readily at room temperature. Similarly, except for $Cu(hfa)_2$, these compounds did not hydrate further at room temperature in a 35% relative humidity environment.

Appendix

Figure 1 represents a simplified schematic of the Multipycnometer. A working equation for the sample volume, V_{samp}, (from which the density can be obtained) can be determined by measuring the pressure difference that results when helium under a known pressure is allowed to flow from a precisely known reference volume, V_{ref}, into a precisely calibrated sample cell, V_{cell}, containing the solid sample, V_{samp}.

The initial states for the reference and the sample volumes can be defined by two independent equations. The reference volume is pressurized with helium whereas the sample cell, V_{cell}, is evacuated and then filled with helium to ambient pressure prior to measurement. The reference volume, V_{ref}, filled with helium at a pressure, P_{ref}, can be defined by the ideal gas equation:

$$P_{ref}V_{ref} = n_{ref}RT \qquad [1]$$

where n_{ref} is the number of moles of pycnometer gas present in the reference cell, at ambient temperature, T, and the measured pressure employed. R is the gas constant in any units desired. The sample cell, V_{cell}, which has an "empty volume", V_{cell}, contains the sample of volume, V_{samp}, and pycnometer gas at ambient pressure, P_{amb}, prior to measuring the densities. The number of moles, n_a, of pycnometer gas at

this ambient pressure is defined in terms of V_{cell}, V_{samp}, and P_{amb} via Eq. [2]:

$$P_{amb}(V_{cell} - V_{samp}) = n_aRT. \qquad [2]$$

The helium in the reference volume at a measured pressure greater than the ambient pressure is then allowed to expand into the sample cell resulting in a new pressure, uniform throughout the system. This pressure, P_{exp}, is defined by,

$$P_{exp}(V_{cell} - V_{samp} + V_{ref}) = n_aRT + n_{ref}RT \ . \qquad [3]$$

Substituting n_aRT from Eq. [2] and $n_{ref}RT$ from Eq. [1] into the righthand side of Eq. [3] yields

$$P_{exp}(V_{cell}-V_{samp}+V_{ref}) = P_{amb}(V_{cell}-V_{samp}) + P_{ref}V_{ref}. \qquad [4]$$

Solving for $(V_{cell} - V_{samp})$ gives:

$$V_{cell} - V_{samp} = \{(P_{ref} - P_{exp})/(P_{exp} - P_{amb})\}V_{ref}. \qquad [5]$$

The pressures on the right hand side of Eq. (5) contains experimentally determined parameters. Since the instrument can be nulled to P_{amb}, the latter drops out of consideration, and we have after rearranging,

$$V_{samp} = V_{cell} - V_{ref}[(P_{ref}/P_{exp}) - 1] \qquad [6]$$

as the volume occupied by the sample. V_{cell} and V_{ref} are calibrated, known values, determined prior to making sample measurement, and P_{ref} and P_{exp} are determined experimentally. The density can be determined by dividing the sample weight by the calculated volume of the sample,

$$\text{Density} = (\text{sample wt})/V_{samp}. \qquad [7]$$

Acknowledgment

This work was supported in part by the Office of Naval Research.

Table I. Chemical analysis of the metal-organic compounds.

Compound	%C	%H	%M	Compound	%C	%H	%M
$Al(acac)_3$				$Al(hfa)_3$			
Theory:	55.55	6.53	8.32	Theory:	27.80	0.47	4.16
actual:	55.19	6.17	8.67	actual:	27.69	0.38	3.92
$Cr(acac)_3$				$Cr(hfa)_3$			
Theory:	51.57	6.06	14.88	Theory:	26.76	0.45	7.72
actual:	51.18	5.69	14.89	actual:	26.70	0.35	7.37
$Co(acac)_2 \cdot H_2O$							
Theory:	43.65	5.86	21.42				
actual:	43.08	5.68	21.38				
$Co(acac)_3$				$Co(hfa)_3$			
Theory:	50.57	5.94	16.54	Theory:	26.49	0.44	8.66
actual:	50.72	5.67	15.99	actual:	26.58	0.35	8.61
$Cu(acac)_2$				$Cu(hfa)_2 \cdot H_2O$			
Theory:	45.88	5.39	24.28	Theory:	24.23	0.81	12.82
actual:	45.96	5.18	23.86	actual:	24.82	0.79	12.47
$Mg(acac)_2 \cdot 2H_2O$				$Mg(hfa)_2 \cdot 2H_2O$			
Theory:	46.45	7.02	9.40	Theory:	25.32	1.27	5.12
actual:	46.48	6.64	9.33	actual:	25.50	1.25	4.67
$Ni(acac)_2$				$Ni(hfa)_2 \cdot 2H_2O$			
Theory anhy	46.75	5.49	22.85	Theory:	23.60	1.19	11.53
actual:	43.71	5.55	22.88	actual:	23.78	1.07	11.30
Theory H_2O:	43.68	5.86	21.35				
$Pd(acac)_2$				$Pd(hfa)_2$			
Theory:	39.43	4.63	34.93	Theory:	23.07	0.39	20.44
actual:	38.97	4.33	34.32	actual:	22.61	0.38	20.55
$Pt(acac)_2$				$Pt(hfa)_2$			
Theory:	30.54	3.59	49.60	Theory:	19.72	0.33	32.02
actual:	30.77	3.41	49.60	actual:	20.11	0.56	-
$Rh(acac)_3$							
Theory:	45.01	5.29	25.71				
actual:	44.66	5.10	25.01				

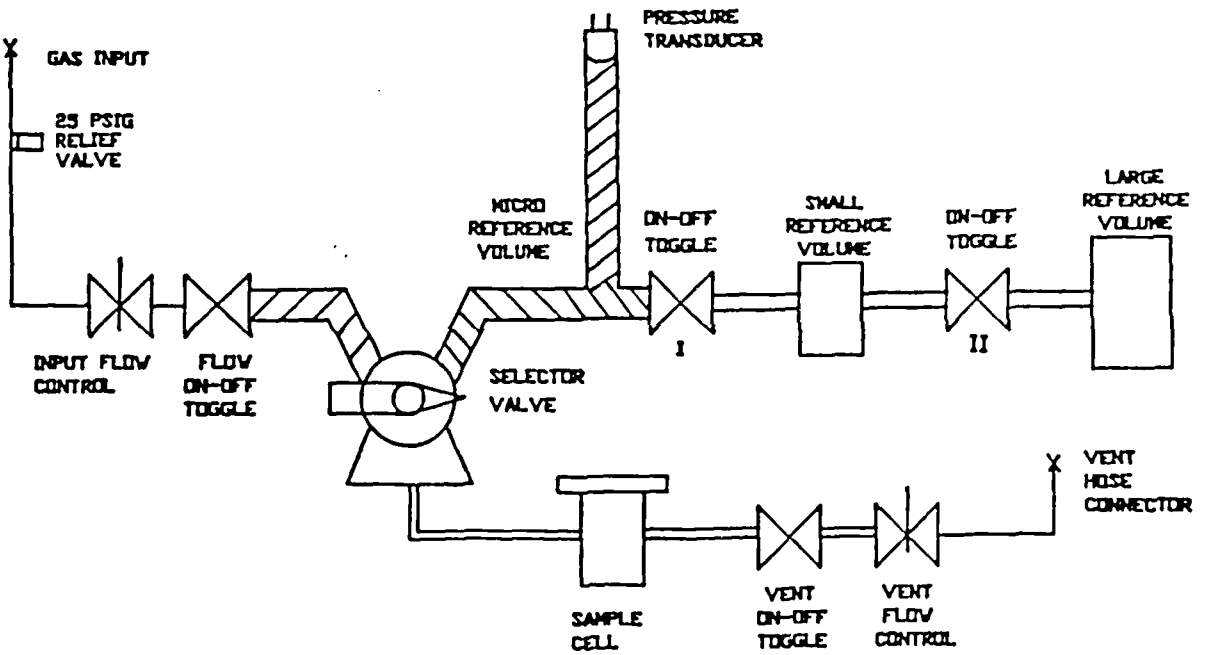
Table I continued

Compound	%C	%H	%M	Compound	%C	%H	%M
$\underline{Zn(acac)_2}$				$\underline{Zn(hfa)_2 \cdot 2H_2O}$			
Theory $2H_2O$:	40.09	6.05	21.82	Theory:	23.30	1.17	12.68
Theory H_2O:	42.65	5.73	23.22	actual:	23.95	1.14	12.94
actual:	41.25	5.27	25.16				
Theory anhy:	45.57	5.35	24.80				
Silver neodecanoate							
Theory:	43.03	6.86	38.64				
actual:	43.12	6.55	38.07				
$\underline{W(CO)_6}$							
Theory:	20.48	na	52.20				
actual:	20.40	na	52.43				

Table II. The densities of the metal-organic compounds.

Compound	Density (g/cm^3)	Literature value (g/cm^3)
0. Al_2O_3	3.994 ± 0.004	3.99 [3]
1. $Al(acac)_3$	1.279 ± 0.000	1.27 [3]
2. $Al(hfa)_3$	1.859 ± 0.003	
3. $Cr(acac)_3$	1.359 ± 0.001	
4. $Cr(hfa)_3$	1.955 ± 0.003	
5. $Co(acac)_2 H_2O$	1.463 ± 0.001	
6. $Co(acac)_3$	1.418 ± 0.000	
7. $Co(hfa)_3$	2.034 ± 0.006	
8. $Cu(acac)_2$	1.594 ± 0.004	1.57 [4]
9. $Cu(hfa)_2 \cdot H_2O$	2.102 ± 0.001	
10. $Mg(acac)_2 \cdot 2H_2O$	1.356 ± 0.003	
11. $Mg(hfa)_2 \cdot 2H_2O$	1.854 ± 0.002	
12. $Ni(acac)_2 \cdot xH_2O$	1.481 ± 0.000	
13. $Ni(hfa)_2 \cdot 2H_2O$	1.971 ± 0.001	
14. $Pd(acac)_2$	1.862 ± 0.005	
15. $Pd(hfa)_2$	2.404 ± 0.022	
16. $Pt(acac)_2$	2.336 ± 0.001	
17. $Pt(hfa)_2$	2.660 ± 0.003	
18. $Rh(acac)_3$	1.607 ± 0.000	1.75 [1]
19. $Zn(acac)_2 \cdot xH_2O$	1.603 ± 0.002	
20. $Zn(hfa)_2 \cdot 2H_2O$	2.003 ± 0.003	
21. Silver Neodecanoate	1.546 ± 0.005	
22. Tungsten hexacarbonyl	2.671 ± 0.002	2.65 [3]

Figure 1. Simplified schematic of the Multipycnometer by Quantachrome.



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